

Serial No.: 10/574,175

Docket No.:

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF:

GROUP:

SERIAL NO:

EXAMINER:

FILED:

FOR:

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes Dr. Hans Görlitzer who deposes and states that:

1. I am a graduate of Technical University Munich received my Ph.D. degree in the year 2000.
2. I have been employed by Röhm GmbH for 5 years as a R&D Manager in the field of Polymer Research.
3. The following experiments were carried out by me or under my direct supervision and control.

The following experiment have been performed to show that binders according to the invention with polydispersity indices below 1.9 are unexpectedly superior to the cited prior art.

The experiments show that the claimed reactive hot melts are superior regarding handling, storage stability and open time.

Measurement of the dynamic viscosity

The dynamic viscosity is measured in accordance with DIN ISO 3219 at 130 °C.

The number average molecular weight M_n , the weight average molecular weight M_w and the molecular weight distribution D are measured by means of gel permeation chromatography (GPC) against a polystyrene calibration.

Hydroxyl numbers were determined potentiometrically.

Example 1

A jacketed vessel equipped with stirrer, thermometer, reflux condenser, nitrogen inlet tube and dropping funnel was initially charged under an N_2 atmosphere with 227 g of methyl methacrylate, 454 g of n-butyl methacrylate, 18.9 g hydroxy ethyl methacrylate, 700 mL toluene, 2.1 g of copper(I) oxide and 5.1 g of PMDETA (pentaethyldiethylene triamine). The solution was stirred at 60°C for 15 min. Subsequently, 5.55 g of w,w-dichloroacetophenone (Aldrich) was added at the same temperature. The mixture was stirred at 80°C for a polymerization time of 5 hours. After introducing atmospheric oxygen for approx. 5 min to terminate the reaction, 8 g of Tonsil Optimum 210 FF (from Südchemie) and 4% by weight of water were added to the solution which was stirred for 60 min. The filtration was effected by means of an elevated-pressure filtration through a filter (Seitz K 700 (700 μ) Mat. B 08582). Toluene and other volatile compounds were evaporated in vacuum.

The mean molecular weight and the molecular weight distribution were finally determined by GPC measurements vs. a poly styrene standard. The copper content of a dried sample of the filtrate was then determined by means of AAS (atom absorption spectrometry).

Example 2

Analogous synthesis to example 1 with 228 g of methyl methacrylate, 456 g of n-butyl methacrylate and 16.1 g hydroxy ethyl methacrylate.

Example 3

Analogous synthesis to example 1 with 230 g of methyl methacrylate, 460 g of n-butyl methacrylate and 9.8 g hydroxy ethyl methacrylate.

Example 4

Analogous synthesis to example 1 with 225 g of methyl methacrylate, 451 g of n-butyl methacrylate and 24.2 g hydroxy ethyl methacrylate.

Reference example 1

A jacketed vessel equipped with stirrer, thermometer, reflux condenser, nitrogen inlet tube and dropping funnel was initially charged under an N₂ atmosphere with 227 g of methyl methacrylate, 454 g of n-butyl methacrylate, 18.9 g hydroxy ethyl methacrylate and 700 mL toluene.

303 g of n-butyl methacrylate, 151 g of methyl methacrylate and 12.6 g of hydroxyl ethyl methacrylate were mixed to form a monomer stock solution. 100 g of the monomer stock solution and 140 g of toluene were charged to a 2 l jacketed reactor with blade stirrer, reflux condenser and nitrogen blanketing. The mixture was heated to an internal temperature of 60°C with stirring. 0.85 g of Peroxan PND from Pergan GmbH, in solution in 5 g of toluene was added. Following the exothermic reaction, the remaining monomer stock solution was metered in together with 3.4 g of Peroxan PND over the course of 3 hours at an internal temperature of 60°C. After the end of the metering, initiation was repeated with 1.4 g of Peroxan PND in 100

g toluene. The subsequent reaction time was three hours. The batch was then cooled to approximately 40°C. Toluene and other volatile compounds were evaporated in vacuum.

	molecular weight M_n [g/mol]	molecular weight M_w [g/mol]	molecular weight distribution (M_w/M_n)	hydroxyl number [mg KOH/g]
Example1	20200	22200	1.10	11.8
Example2	19700	22000	1.12	9.1
Example3	19100	21700	1.14	6.4
Example4	20100	22700	1.13	15.6
Comparative example 1	18500	37300	2.02	6.1

Preparation of reactive hot melts

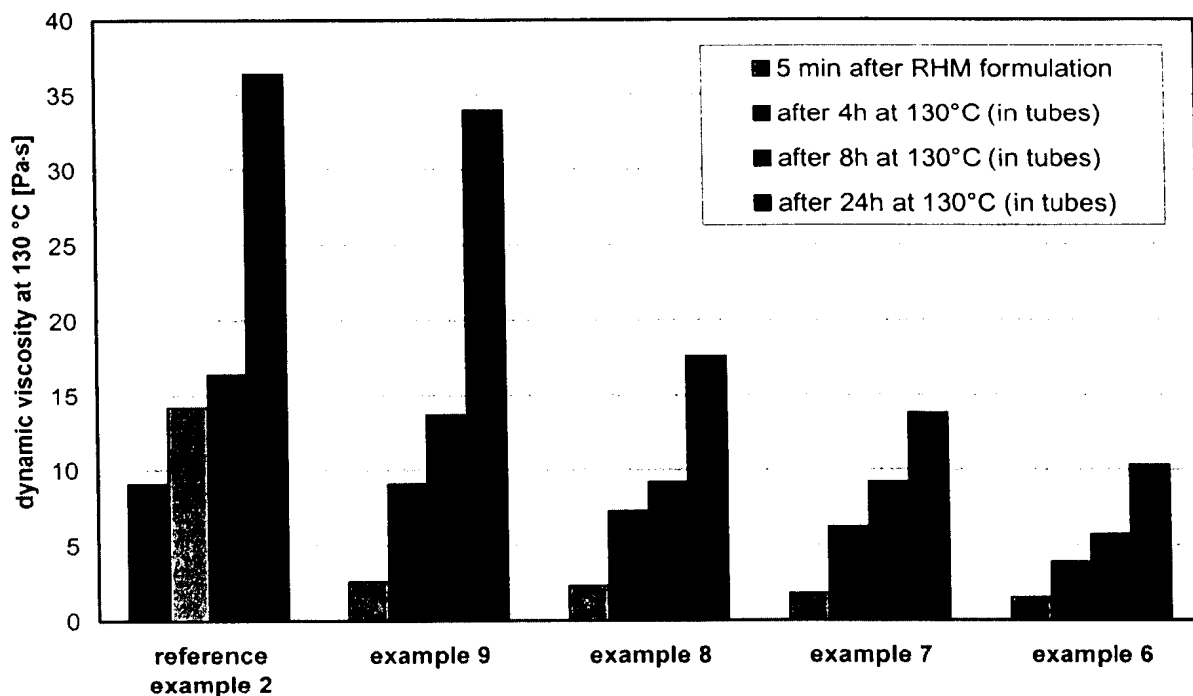
88.4 g of a poly propylene oxide with a hydroxyl number of 56 mg KOH/g (Voranol 2000 L by Dow chemical cooperation), 35.4 g of a polyester with a hydroxyl number of 28.8 mg KOH/g (Dynacoll 7360 by Evonik Degussa GmbH) and 49 g of one of the methacrylic examples 1 to 5 as described before were melted at approximately 180 °C in a 500 mL flask. The homogeneous formulation was cooled down to 130 °C and evaporated in vacuum. 24 g of an isocyanate (Suprasec 3051 (08/03) by Huntsman cooperation) were added and the mixture was stirred for another 45 minutes at 130 °C under an N₂ atmosphere. The dynamic melt viscosity of the reactive hot melt was measured. Additional samples were filled in 100 mL

tubes and stored at 130 °C. The dynamic melt viscosities were measured again after 4, 8 and 24 hours.

	methacrylate component	dynamic viscosity after 5 min [Pa*s]	dynamic viscosity after 4 hours [Pa*s]	dynamic viscosity after 8 hours [Pa*s]	dynamic viscosity after 24 hours [Pa*s]
Example 6	Example 3	1.5	3.9	5.7	10.3
Example 7	Example 2	1.8	6.2	9.2	13.8
Example 8	Example 1	2.3	7.3	9.2	17.6
Example 9	Example 4	2.6	9.1	13.7	34.0
Reference example 2	Reference example 1	9.1	14.2	16.4	36.4

The reference sample 1 shows the lowest number average molecular weight of all methacrylic binders in this series. Nevertheless the reactive hot melt based on this sample (reference sample 2) had the highest dynamic viscosity after preparation. Respectively the dynamic melt viscosity after storage of 24 hours was comparable to a binder with about three times more hydroxyl groups (example 5 in formulation of example 9) that was prepared according to the invention. Typically should the melt viscosity depend on the number of polar – e.g. hydroxyl - groups. Surprisingly was the molecular weight effect in this case of more importance.

It is show that the polymers with molecular weight distributions (mwd) below 1.9 were superior to broad distributed binders (mwd > 1.9) with identical composition, hxdroxyl number and number average molecular weight. For this reason these novel reactive hot melts according to the invention are superior to the prior art regarding handling, storage stability and open time.



4. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

5. Further deponent saith not.

Customer Number

22850

Tel. (703) 413-3000
Fax. (703) 413-2220
(OSMMN 05 06)

Signature

Date

[Handwritten Signature]

April 22, 2009